

PATENT SPECIFICATION

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COMPLETE SPECIFICATION

Cracking Heavy Hydrocarbon Oils to Produce Olefins, Motor Fuels and Coke

We, ESSO RESEARCH & ENGINEERING COMPANY, a corporation duly organized and existing under the laws of the State of Delaware, United States of America, having an office at Elizabeth, New Jersey, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention is concerned with a process and apparatus for high temperature coking of heavy hydrocarbon oils to produce motor fuels, coke, and unsaturated hydrocarbons of low molecular weight. The invention is more particularly concerned with a process and apparatus wherein finely divided preheated solid particles which are relatively inert catalytically are employed to accomplish the desired conversion.

In the prior art a number of suggestions have been made for converting heavy hydrocarbonaceous oil to the relatively valuable olefins and diolefins having 2 to about 6 carbon atoms. The process also is adapted to the production of benzene and homologous aromatic hydrocarbons as well as to the production of paraffinic hydrocarbons and hydrogen. In recent years demands have increased for such products as ethylene, propylene, butadiene, isoprene, and benzene. These materials are particularly useful as starting materials for production of various chemicals including polymeric materials. Various plastics, and synthetic fibers, as well as simpler chemical reagents and solvents may be derived from these materials. Hence, the low molecular weight unsaturates may be considered as chemical building blocks for the production of numerous products of industry including synthetic fibers, plastics and other polymers.

It has long been known that by cracking heavy hydrocarbon oils, such as petroleum residua, and coal tar residua, various unsaturates may be obtained in substantial quantities.

[Price 3s. 6d.]

According to one process preheated solid particles are used to contact the feed which is converted thermally to the desired products. The desired products are separated from the solid particles and quenched by contact with a cooling fluid. The separated solids are recycled and reheated to maintain heat requirements in the system.

The present invention is analogous in several respects to the above process but represents a number of improvements over it. In accordance with the invention, there is provided a process for converting heavy hydrocarbonaceous oil to produce more volatile products including substantial proportions of C_2 — C_4 olefins, plus coke, which process comprises passing a stream of hot inert solid fluidizable particles, preheated to a temperature above about 1300°F., and gas-suspended to an apparent density of about 0.5 to 10 pounds per cubic feet, into and out of an elongated coking zone, maintaining a total pressure of from 12 to 50 p.s.i.a. in the coking zone, feeding the oil to be converted in finely divided but predominantly liquid phase into contact with the hot particles, feeding a non-hydrocarbon and substantially non-reactive gas into the coking zone to maintain particle suspension and also to reduce hydrocarbon partial pressure, the oil feed and the flow rate of the non-reactive gas being adjusted to keep the hydrocarbon partial pressure below 20 p.s.i.a. and below 85% of the total pressure, and the oil particles' average contact time in the reaction zone below 2 seconds but sufficient to obtain relatively large yields of C_2 — C_4 olefins and/or diolefins and to deposit a carbonaceous residue on the particles, passing the resulting mixture of vapours and gas with entrained deposit-bearing particles into a quick-acting separation zone to remove the entrained particles, and immediately quenching the vapours by cooling them by at least 200 Fahrenheit degrees, the separation and quenching being accomplished within an average time

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Price 4s 6d

of less than two seconds.

The invention also provides apparatus for coking heavy hydrocarbonaceous oil to produce more volatile products and coke, which comprises a transfer line adapted to carry a stream of suspended particles, means for spraying oil in finely divided form into contact with a stream of fluidizable solids flowing in the transfer line, means for supplying a suspending and diluting gas to assist in carrying the particles through the transfer line, means for adjusting the feed rate of the oil and of the diluting gas to the reactor, a rapid gas-solids separating cyclone at the outlet end of the reactor comprising an outer downwardly converging wall defining the cyclone vessel and an inner downwardly extending perforate frusto-conical baffle connected to an upflowing vapour outlet line, a downwardly extending solids outlet stripper vessel, means for stripping vapours from solids flowing down the stripper vessel, means for reheating the separated solids, and means for returning the reheated solids to the reactor to supply heat to it.

By proceeding in accordance with the invention, the efficiency of conversion is improved and high economic yields of desirable low molecular weight products can be obtained from heavy oils which are of low economic value.

The conversion of the heavy oil is preferably carried out at temperatures of 1250—1400°F., and a particularly suitable operating temperature for the overall production of a range of desirable low molecular weight products is in the neighbourhood of 1300°F. As the diluent, superheated steam is preferred, although nitrogen, flue gas and other inert diluents may be used.

As stated above, the invention contemplates an average reaction time, that is, the time that the oil is in contact with the heat carrying particles, which is very short. The preferred contact time is in the general range of about 0.5 seconds for a temperature of 1300°F. At a moderately lower temperature, the time preferred is a little longer, for example, about 1 second at 1275°F. At higher temperatures the contact time ordinarily should be shortened.

The invention also involves the use of highly efficient vapor-solids separating equipment in order to hold the separation and quenching time to or near a minimum. The separation time should not exceed 1 second, and preferably is less. It should be shorter, as a rule, than the reaction time, and preferably not longer than about 0.1 to 0.25 seconds in the preferred operating temperature range of 1275—1350°F. At higher temperatures the separation time should be even shorter. It is necessary to cool the separated vapors by at least 200 Fahrenheit degrees in one second, and it is preferable to cool them more, *i.e.* several hundred degrees, *e.g.* down to 600—

700°F. to prevent thermal degradation of products.

A further feature of the invention involves heating the solids which supply heat for the conversion by burning combustible material in the presence of the solids in a very short time period. It has been found that if the burning period is prolonged, the carbonaceous residues deposited on or constituting the solid react with carbon dioxide produced by the combustion to produce carbon monoxide with considerable loss in thermal efficiency. It is important, therefore, that the reheating of the solids be accomplished by burning during a period of less than 3.0 seconds and preferably less than 1.5 seconds. In this way the production of carbon dioxide is maximized and a good heat economy is obtained. Where possible it is highly desirable to keep the combustion time affecting a particular particle below 1.0 seconds.

Other features include an improved recovery and fractionating system and controls by which the production of dry gases, *i.e.* hydrocarbons of 3 carbon atoms and less, is kept between about 25 and 40% by weight of the oil feed. The preferred conversion to dry gas is about 30%.

The invention will be more clearly understood by reference to the accompanying drawings wherein Fig. 1 is an elevational view, with certain parts omitted, of a coking system for carrying out the process of the invention.

Fig. 2 is an enlarged view of the reactor, separator and quenching elements shown in the central part of Fig. 1, certain parts being omitted and others shown in cross-section.

Fig. 3 is a graph showing the effect of hydrocarbon partial pressure on various products obtained in thermal cracking at high temperatures.

Referring now to the drawings, the apparatus will be described in detail. A stream of preheated solids at a temperature preferably above 1400°F. is supplied by a standpipe 11 through a reverse bend or U-bend 13 into an upflowing leg or reverse standpipe 15 from which it flows into an elongated reaction vessel 17. The velocity of flow through the reactor and the size of the reactor are so proportioned that the average particle passes entirely through the reactor in a time not greater than 2 seconds. The preferred velocity is such that the average particle will pass through the reactor in about 0.5 seconds and it may be less at higher temperatures. For a reactor temperature of 1300°F. a superficial velocity for the stream of solid particles is preferably 10 to 50 feet per second for a reactor 20 to 50 feet long.

The standpipes just described are aerated and the solids are propelled by injection of steam through one or more of a set of nozzles 19, 21 and 23. Additional fluidizing gas, preferably steam, though other inert gases men-

tioned above may be used, is introduced into the reactor 17 through an inlet line 25. By adjusting the flow of steam in the several lines just mentioned, the proper velocity of solids flow as well as the proper hydrocarbon partial pressure may be obtained in the reactor.

The oil to be converted is introduced through a line 27 into a nozzle or multiple nozzle arrangement 29 which is preferably in the form of an annulus so as to distribute the oil feed quite uniformly over the transverse area or cross section of the reactor. In this manner, the stream of hot particles is uniformly contacted with the feed. The feed which may be preheated but is largely or substantially in liquid phase, is sprayed forceably through the nozzle device 29 to achieve good dispersion and to break it down into finely divided droplets or particles.

In a typical operation, the temperature of the solid particles entering the reactor from line 15 may be about 1400°F. At initial contact with the oil, the temperature in the reactor may be about 1370°F. whereas in the reactor outlet the temperature will be substantially lower; for example, about 1290—1300°F.

The reactor terminates in a cyclone of special design which is shown more fully in Fig. 2.

Referring to Fig. 2 it will be seen that the reactor terminates in a downwardly spiralling and converging exterior body member 31. This forms the outer wall of the cyclone or separator. A view looking down on the apparatus of Figure 2 would show the incoming stream of solids and vapor rotating, e.g. clockwise, and converging as it moves downwardly. Within the member 31 is a frusto-conical element 33 formed as the terminal lower portion of outlet line 35. The terminal frusto-conical portion is perforated with a large number of holes, which are large enough to permit ready escape of gas and vapor as indicated at 37.

Due to centrifugal force the solid particles flow around the inner periphery of the outer wall member and then downwardly. The vapors pass quickly through the perforations and upwardly through outlet line 35 to recovery apparatus to be described.

A quenching fluid is introduced through a line 39 to a manifold or annular member 41 designed to direct a spray of quenching material into intimate contact with the outflowing vapors. Preferably the spray is directed upwardly as shown but it may be directed downwardly. Also, the outlet line may pass downwardly if desired. A branch line of the quench outlet extends downwardly as indicated at 43 to or nearly to the bottom of the frusto-conical element 33. This is provided with a number of branch nozzles directed upwardly so that a cooling spray may be introduced into the vapors just as soon as they come into the outlet line. These nozzles are shown at 45.

While the arrangement may be varied somewhat a prompt and effective quenching is necessary to prevent degradation of the vapor products.

The quenching material may be a cooling liquid such as water or oil, or it may be a suspension of cool solid particles in a suitable fluid as is known in the art. Preferably, however, an oil of intermediate boiling range between the low molecular weight products and the original feed is used for quenching. For example, a particularly suitable quenching liquid may be an oil cut of boiling range above about 400°F. up to 750°F. or so. An oil boiling up to 900°F. may be used in most cases but the preferred quench oil is not quite so heavy. It is injected at a sufficient rate to bring the temperature of the product vapors down by at least 200° and preferably not less than 250°F. within 1 second. Preferably the quenching is accomplished within a much shorter period, for example, 0.25 seconds or less, as indicated above. It is preferred also, as indicated above, to quench even more drastically by bringing down the temperature all the way to 800°F. and preferably to 700°F. or less from its original temperature of 1250°F. or higher.

Returning now to Figure 1, the quenched vapors, which are now relatively inert as regards auto degradation, are carried through a line 51 to recovery apparatus 53. The latter comprises a series of baffles 55 in its lower portion for scrubbing the quenched vapors as they pass upwardly. Heavy condensed oil may be withdrawn from the bottom of tower 53 through outlet line 57. This oil, or a portion thereof, may be recycled to the coker reactor 17 if desired.

The uncondensed vapors pass upwardly through a series of plates 59 for further fractionation, the condensed liquid therefrom collecting on one of the plates 61 for removal through line 63. This liquid may be returned by a conventional pumparound system through line 65 above, so as to flow down countercurrent to the rising vapors and thus cool and condense them more effectively. The vapors which rise above the line 65 are further fractionated in the upper portion 67 of the tower which contains suitable fractionating plates 69. Various side streams may be withdrawn as desired. One of these, preferably of intermediate boiling range, may be used as a quench liquid at nozzles 41, 45. The uncondensed residual gases are taken overhead through outlet line 71.

During the conversion in the reactor carbonaceous deposits, mainly of coke but usually containing some oil which continues to evolve vapors, are deposited upon the original particles fed to the reactor. As these deposit-bearing particles are separated from the vapors, they are passed downwardly into a collecting zone 81. Here they may be stripped by a strip-

ping gas introduced through line 83 so as to recover additional vapors. The latter may be passed upwardly to the product outlet line in counter flow to the downflowing solids. Alternatively, if desired, they may be isolated by a vapor seal established at the narrow section 85. Preferably, the velocity and volume of gas introduced through line 83 is sufficient to maintain a fluidized bed of solids within the collecting vessel 81.

The stripped solids are passed from the collection vessel through an outlet line 89 and reverse or U-bend 91 into the upflowing line 93. Suitable aeration and propulsion jets, preferably of steam, are provided at intervals as indicated at 95, 97 and 99. Where it is desired to reduce the particle size of the solids these inlet lines may be in the form of high velocity jets. The use of such jets, i.e. at velocities of several hundred feet per second, breaks up the solid particles quite effectively, especially when these are of product coke as is usually preferred. The rate of flow of solids through line 93 is controlled by adjusting the quantity of gas flow through jets 95, 97 and 99.

A valve 101 is provided for safety shut-off. In Fig. 1 it is shown near the top of line 93 but it can be near the bottom thereof if desired, or in the lower portion of standpipe 89 as will be obvious.

The upflowing solids which have been stripped of their hydrocarbon residue and are therefore low in volatiles content are passed into a large elongated burning vessel 105. A stream of combustion supporting gas, such as air or oxygen, is supplied to line 105 through inlet 107. If the particles themselves are not combustible and/or do not contain enough carbon to supply heat requirements, extraneous fuel may be added along with the air. Even when the particles are coke, as usually preferred, they need not be burned if it is desirable to obtain product coke. In this case, all of the required fuel, e.g. oil or gas, will be supplied along with the air to line 107 or some of it may be added at subsequent points in the burner if desired. The extraneous fuel burns preferentially, leaving the product coke largely unburned.

As noted above, it is desirable to complete the combustion and reheating of the solids quickly. This is an important subsidiary feature of the present invention. Accordingly, the burner is so proportioned that the average particles pass completely through it in less than about 3.0 seconds and, preferably, in less than 1.5 seconds. Thus, for a burner vessel of 60 feet in length the average velocity should be at least 20 feet per second and preferably somewhat greater. Because of this velocity, it is desirable to use an effective but quick separating cyclone for separating the solids from the combustion gases. Such a cyclone is indicated at 109 and it is of the same

general construction as cyclone 31, 33 described above.

The solids are separated from the combustion gases and the latter are passed overhead through outlet line 111. Since this is a quick separation cyclone, it is usually desirable to pass the combustion gases through a secondary cyclone to further recover entrained fine particles and prevent objectionable atmospheric pollution. The secondary recovery means not shown, may be of any conventional type.

The separated solids pass downwardly into stripping and collecting vessel 113 which operates in somewhat analogous fashion to vessel 81, previously described. Stripping gas may be introduced through one or more inlets 115, 117 and the stripped reheated particles flow down the standpipe 11 and are recycled to the reactor to continue the process. Product coke may be withdrawn at any suitable point in the system, for example, through the outlet line 117' under control of valve 119 in the standpipe 11. Under some conditions of operation, the coke produced in the process is completely consumed so that there is no by-product coke.

Referring now to Fig. 3, which is concerned with a coking zone operating at a total pressure of 25—30 psia, it will be seen that the production of the most desirable vapor products increases very markedly at low hydrocarbon partial pressure. At the same time, the production of methane, which is usually of low economic value and hence undesirable as a major product, drops off rapidly below 10 p.s.i.a. hydrocarbon partial pressure. It is highly desirable from a yield standpoint, therefore, to operate at a hydrocarbon partial pressure below 10 psi. Of course, the quantity of inert diluent is determined by the desired hydrocarbon partial pressure. For example, the following tabulation shows the weight per cent steam on feed required for hydrocarbon partial pressures of 5, 10 and 20 psi.

Hydrocarbon partial pressure, psi	5	10	20
Wt. % Steam	130	48	6.6

The optimum quantity of steam to use depends upon the relative values of steam and hydrocarbon products and equipment costs at the particular location at which the process is used.

The correlations shown in Fig. 3 give the total C_1 , C_2 , C_3 and C_4 yields obtained when feeding gas oil to the reactor. Although the yields obtained with the residua feed stocks differ somewhat from those shown in Fig. 3, the data available indicate that the trends with hydrocarbon partial pressure are about the same. The yields shown in Fig. 3 include both saturated and unsaturated hydrocarbons. Under the preferred operating conditions of

temperature and contact time described above, namely, about 1300°F. and preferably around 0.5 seconds or less, the C₂ products yield 60% or more of ethylene. The C₃ products are usually over 90% olefinic and the butadiene, which is the most valuable of the C₄ products, usually runs between 30 and 40% of total C₄ under these preferred operating conditions.

As noted above, the conversion to dry gas (i.e. C₁ and lighter) determines to a considerable degree the overall efficiency of the process. It is preferred to keep this dry gas conversion between the limits of 25 and 40% by weight, based on the feed. The conversion of about 30% for most feeds is close to the optimum. It will be understood also that other desirable products than those specifically mentioned above are obtained under the same operating conditions. The C₂ diolefins are obtained in reasonably good yields and some yields of benzene, toluene and xylene are obtained, all of which are of greater value than gas oil or gasoline in most refineries.

As a specific example of a satisfactory operation, solid particles preheated to 1500°F. are fed into the reactor along with superheated steam. The standpipe velocity prior to steam introduction is between about 3 and 5 feet per second. The addition of steam increases this to 15 to 17 feet per second. The residuum feed, for example a reduced Louisiana crude, is sprayed through the distribution nozzle or ring 29 into the hot solids. The feed produces sufficient gas upon vaporization and cracking to raise the velocity to 60 feet per second at the reactor outlet. The nominal gas time in the transfer line portion of the reactor is about 0.25 seconds. In the cyclone the time of reaction is about 0.25 seconds. The cyclone is the terminal portion of the reactor in effect and it is important that, as far as possible, the gas be withdrawn from all contact with the solids as rapidly as it is separated. The product vapors are quenched at the cyclone outlet by a spray of cold oil, and cooled down to 540°F. to quench the more volatile vapors and to condense a fraction of the 650°F.+ tar in the products. The quench oil used here is a 430° to 650°F. cut, recycled from the fractionator. The solids which are separated from the products are stripped with steam in a stripping section having a solids hold-up time of about 10 seconds. A steam rate giving 2 V/V in the stripper is sufficient to prevent appreciable loss of vapor products. Otherwise, 2 or 3% of the product vapors would be carried into the burner.

Upon entering the burner, the stripped solids are mixed with air, and the combustion proceeds. The temperature of the solids reaches 1500°F. within 1.5 to 2 seconds or so, and the burner is designed for an outlet velocity of 60 feet per second. The heated solids are separated from the flue gases in a

short-time cyclone 109. The waste heat in the flue gases is recovered in a waste heat boiler not shown. The solids from the short-time cyclone are then dropped into the receiver stripper vessel 113 and are stripped with steam to avoid carrying flue gases into the reactor. With the design shown in Figure 1, some of the stripping steam introduced through line 83 flows upwardly into the outlet line 33—35.

As an example, the following products are obtained when feeding 2.4% South Louisiana residuum at 1300°F., 30% conversion to C₁ and lighter, the conditions being 5 p.s.i.a. hydrocarbon partial pressure, 12 p.s.i.a. total pressure, and about 130 weight per cent steam, based on the hydrocarbon feed:

Product Yields, Wt. %

Non-hydrocarbon gas (excluding H ₂)			
H ₂	0.9		85
CH ₄	0.6		
C ₂ H ₄	6.3		
C ₂ H ₆	10.3		
C ₃ H ₆	2.8		
C ₃ H ₈	8.5		90
C ₄ H ₆	0.6		
C ₄ H ₈	2.24		
C ₄ H ₁₀	4.1		
C ₄ H ₁₂	0.1		
C ₅ —430°F. (excluding benzene)	17.2		95
Benzene	0.83		
430—650°F.	7.2		
650°F.+ (including coke)	38.4		

It will be understood that the example just recited is only one of several modes of operation contemplated. The system is operable within the limits previously described and is highly efficient. Reduced crude of low economic value is converted to products of high value and good yields by operating within the conditions specified.

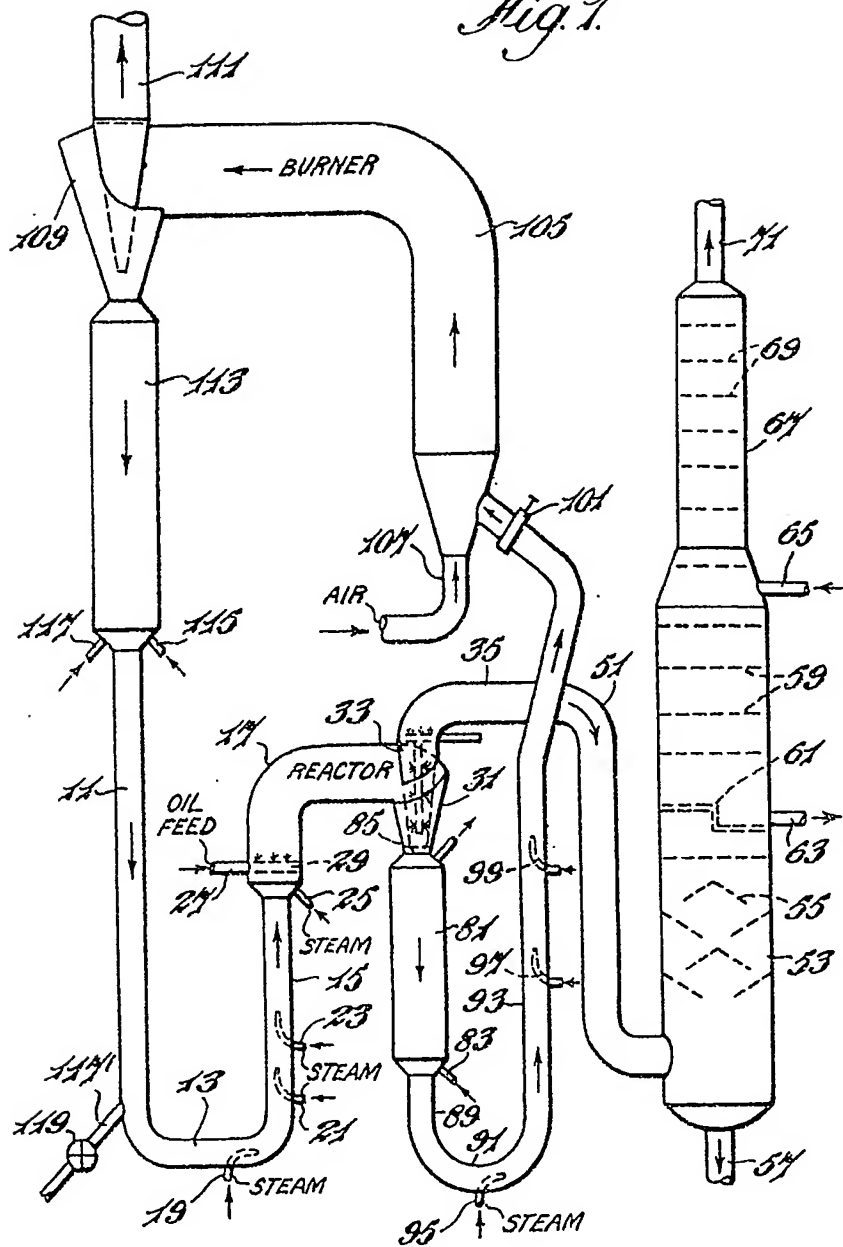
WHAT WE CLAIM IS:—

1. A process for converting heavy hydrocarbonaceous oil to produce more volatile products including substantial proportions of C₂—C₄ olefins, plus coke, which process comprises passing a stream of hot inert solid fluidizable particles, preheated to a temperature above about 1300°F., and gas-suspended to an apparent density of about 0.5 to 10 pounds per cubic foot, into and out of an elongated coking zone, maintaining a total pressure of from 12 to 50 p.s.i.a. in the coking zone, feeding the oil to be converted in finely divided but predominantly liquid phase into contact with the hot particles, feeding a non-hydrocarbon and substantially non-reactive gas into the coking zone to maintain particle suspension and also to reduce hydrocarbon partial pressure, the oil feed rate and the flow rate of the non-reactive gas being

- adjusted to keep the hydrocarbon partial pressure below 20 p.s.i.a. and below 85% of the total pressure, and the oil particles' average contact time in the reaction zone below 2 seconds but sufficient to obtain relatively large yields of C_2 — C_4 olefins and/or diolefins and to deposit a carbonaceous residue on the particles, passing the resulting mixture of vapours and gas with entrained deposit-bearing particles into a quick-acting separation zone to remove the entrained particles, and immediately quenching the vapours by cooling them by at least 200 Fahrenheit degrees, the separation and quenching being accomplished within an average time of less than two seconds.
2. A process according to Claim 1, in which the particles are preheated to a temperature within the range 1300—1800°F.
3. A process according to any one of the preceding claims, in which at least a substantial part of the separated particles are reheated and recycled to the reaction zone to supply necessary heat to it.
4. A process according to Claim 3, in which the reheating is accomplished by partial combustion of the carbonaceous deposits.
5. A process according to Claim 3, in which at least part of the reheating is accomplished in a heating zone by combustion of extraneous fuel in immediate contact with a suspension of said particles.
6. A process according to any one of the preceding claims, in which the non-hydrocarbon gas comprises superheated steam.
7. A process according to Claim 6, in which the non-hydrocarbon gas is superheated steam supplied in proportions of 5—150% by weight based on the oil feed rate.
8. A process according to any one of the preceding claims, in which the reaction zone is maintained at a temperature above 1200°F.
9. A process according to Claim 8, in which the temperature is from 1250 to 1400°F.
10. A process according to Claim 9, in which the coking temperature in the reactor is between 1275° and 1350°F.
11. A process according to any one of the preceding claims in which the total contact time in reaction zone and separation zone is from 0.2 to 2 seconds.
12. A process according to any one of the preceding claims in which reactor contact time is 0.25 to 0.5 second, gas-solids separating time is not more than 0.25 second, and quenching is accomplished by cooling down to a temperature not exceeding 750°F. within 0.25 second after separation of the solid particles.
13. A process according to any one of the preceding claims, in which the total conversion to dry gas (C_2 and lighter) is kept between 25 and 40% based on the weight of the oil feed.
14. A process according to Claim 13, in which the conversion is kept to about 30 wt. %.
15. A process according to any one of the preceding claims, in which the quenching medium is a cool hydrocarbon oil.
16. A process according to Claim 15 in which the quenching oil is an oil cut boiling below 700°F.
17. Apparatus for coking heavy hydrocarbonaceous oil to produce more volatile products and coke, which comprises a transfer line adapted to carry a stream of suspended particles, means for spraying oil in finely divided form into contact with a stream of fluidizable solids flowing in the transfer line, means for supplying a suspending and diluting gas to assist in carrying the particles through the transfer line, means for adjusting the feed rate of the oil and of the diluting gas to the reactor, a rapid gas-solids separating cyclone at the outlet end of the reactor comprising an outer downwardly converging wall defining the cyclone vessel and an inner downwardly extending perforate frusto-conical baffle connected to an upflowing vapour outlet line, a downwardly extending solids outlet stripper vessel, means for stripping vapors from solids flowing down the stripper vessel, means for reheating the separated solids, and means for returning the reheated solids to the reactor to supply heat to it.
18. Apparatus according to Claim 17, in which the reheating means comprises a transfer line burner and a quick-separating cyclone of the same general type as used in the reactor.
19. Apparatus according to Claim 17 in which quenching means are provided in the vapour outlet line for quick quenching.
20. Apparatus for coking heavy carbonaceous oil substantially as described with reference to the accompanying drawings.
21. A process for coking heavy hydrocarbon oil substantially as described.
22. A process as claimed in Claim 1, when carried out in apparatus as claimed in any one of Claims 17—20.

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Fig. 1.



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2 SHEETS

This drawing is a reproduction of
the Original on a reduced scale.
SHEETS 1 & 2

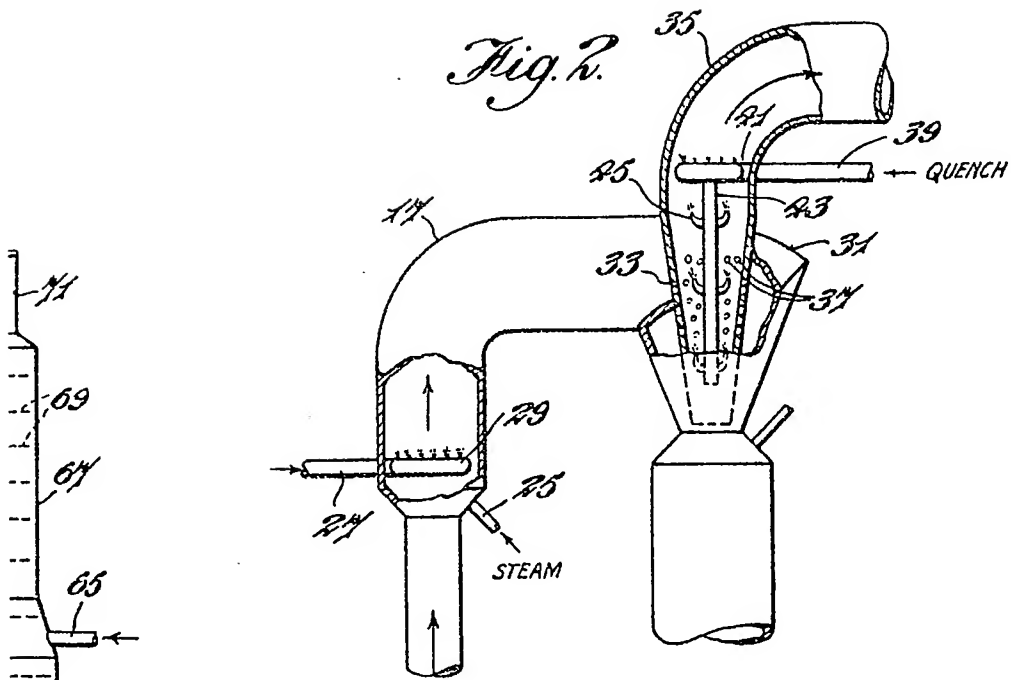
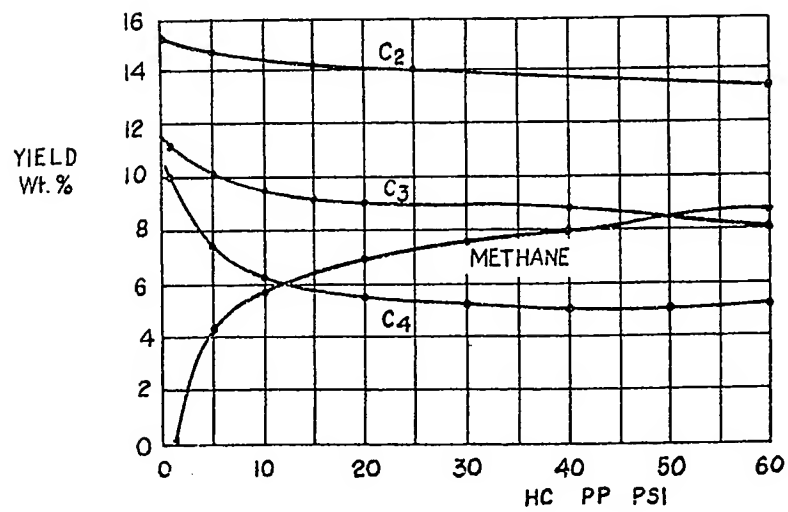


Fig. 3.



POOR QUALITY

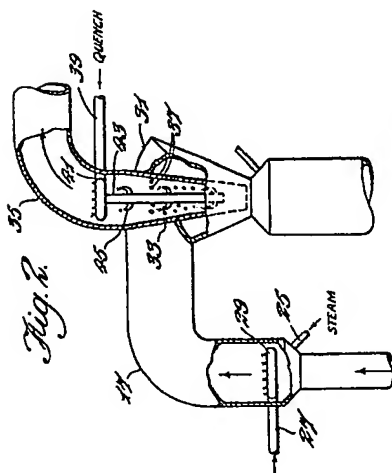


Fig. 3.

